

# PATENT SPECIFICATION

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## (54) PROCESS FOR PREPARING PRILLS FROM A UREA MELT CONTAINING MONOAMMONIUM PHOSPHATE

(71) We, UNIE VAN KUNST-MESTFABRIEKEN B.V., a Netherlands Limited Liability Company, of P.O. Box 45, Utrecht, the Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the prilling of a urea melt containing monoammonium phosphate.

It is known that a urea melt which contains monoammonium phosphate in suspension eg. in an amount of about 50% by weight, cannot be prilled because of gel formation, and to effect prilling it has been proposed to incorporate in the suspension a proportion of potassium metaphosphate. However the presence of potassium metaphosphate does provide some disadvantage in the preparation of urea prills containing monoammonium phosphate. It is also known that prillable urea melts containing monoammonium phosphate can be obtained without the addition of potassium metaphosphate by vigorously stirring the mixture for an extended period of time, eg. 6 minutes or more. However the resulting prills have an insufficient breaking strength resulting in the caking of the prills during storage. In addition such a protracted mixing time causes a rise in the biuret. Such disadvantages are obviated by the present invention, whereby a prillable suspension is obtained and the resulting prills have a good breaking strength.

The invention provides a process for prilling a urea melt containing monoammonium phosphate, wherein the urea melt is mixed with solid monoammonium phosphate less than 10 seconds before effecting prilling of the melt.

The mixing time denotes the period of time that elapses between the time the urea melt is fed into the mixing apparatus and the time the resulting drops are discharged from the prilling apparatus. This period of time can be determined in a simple manner by adding a colouring agent to the urea melt at a particular time during continuous operation, and noting the appearance of coloured prills.

With the short mixing time used in the process of the invention, the formation of biuret and loss of ammonia, which escapes as a gas during the mixing process, appear to be considerably less than when use is made of long mixing times according to previously-described processes. Particularly good results can be obtained if use is made of a mixing time of less than 6 seconds.

The urea melt used in the process according to the invention can be obtained in a known manner by evaporating an aqueous urea solution or by melting so-called crystalline urea. Preferably the water content of the urea melt is below 0.5% by weight so that it is not necessary to dry the prills. The monoammonium phosphate used may be that which is obtained from phosphoric acid prepared by the so-called wet method, or from phosphoric acid prepared by the so-called thermal method. These two methods are for liberating phosphoric acid from raw phosphate. In the "wet" method, the raw phosphate is dissolved by a strong acid e.g. sulphuric acid, and phosphoric acid obtained from this solution. In the "thermal" method, in a preferably electrically heated oven, phosphorus pentoxide is liberated from the raw phosphate, and is converted to phosphoric acid by dissolution in water. The wet type of monoammonium phosphate is preferred as the required rapid mixing of the urea melt proceeds considerably better, under otherwise similar conditions. It is preferred that at least a portion of the monoammonium phosphate e.g. about 15 per cent, used in the process of the invention has been prepared by the wet method.

Other substances may also be added to the urea melt, e.g. diammonium phosphate. Generally the monoammonium phosphate also contains some diammonium phosphate. Examples of other additives which may be incorporated in the melt are ammonium nitrate, potassium chloride, potassium sulphate and magnesium carbonate. These substances may be added separately or mixed with the monoammonium phosphate. The required fineness of the monoammonium phos-

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phate and of other substances which may be added, depends on the particular method of prilling used. If use is made of a prilling head with apertures of for example 1.5 millimeters, a particle size of less than 600 $\mu$  appears to be necessary. If use is made of a rotating disc, larger particles may be used.

The urea content of the prills obtained according to the invention may vary, and is preferably more than 40% by weight.

The mixing of the urea melt with monoammonium phosphate and with any other additions, can readily be effected in a Cyclone mixer, for example in which the melt is fed tangentially to the CYCLONE (Trade Mark) mixer and the solid substance to be admixed introduced centrally after being preheated to 80°C. The resulting suspension can be sprayed by means of a rotating prilling head. After leaving the prilling head, the drops are solidified by cooling and then stored. It is preferred to cool the prills obtained according to the invention to below 35°C before storage to obtain prills of the best possible quality.

The following Examples of the invention are provided:

#### Examples I—V.

A CYCLONE mixer with a diameter of 37 millimetres was fed tangentially with molten urea at a temperature about 135°C and centrally with monoammonium phosphate or a mixture of monoammonium phosphate and potassium chloride at a temperature about 80°C. The resulting suspension was prilled in air by means of a cylindrical prilling head having a height and a diameter of 75 millimetres and provided with a scraper and with

90 spraying apertures of 1.5 millimetres in diameter. The speed of the prilling head was 450 revolutions per minute. The through-put rate was about 120 kilograms of prills per hour and the mixing time about 1.2 seconds. The particle size distribution of the monoammonium phosphate and the potassium chloride was: 28% smaller than 100 $\mu$ , 54% smaller than 150 $\mu$ , 98% smaller than 300 $\mu$ , and 100% smaller than 400 $\mu$ . The molten urea contained about 0.3% by weight of water and the K<sub>2</sub>O content of the potassium chloride was about 60% by weight. Various grades of monoammonium phosphate were used, namely a virtually pure product in Example I (12.2% by weight of N, 61.6% by weight of P<sub>2</sub>O<sub>5</sub>), a strongly contaminated product in Examples II and V (11.8% by weight of N, 54.5% by weight of P<sub>2</sub>O<sub>5</sub>), and a product containing diammonium phosphate in Example III (14.7% by weight of N, 53.0% by weight of P<sub>2</sub>O<sub>5</sub>). The product used in Example IV was composed of 80 parts by weight of the product used in Example I and 20 parts by weight of the product used in Example II. The virtually pure product was obtained from phosphoric acid prepared by the so-called thermal method and the strongly contaminated product from phosphoric acid prepared by the wet method. The accompanying Table shows the analyses of the resulting prills, and also the breaking strength measured directly after the prills had been cooled to 25°C and measured after the prills had been stored at about 25°C for 12 weeks. For comparison, the corresponding data of prilled urea without additions are mentioned under Example A (not according to the invention).

TABLE

Example	I	II	III	IV	V	A
Analysis						
% by weight of N	35	30	29.5	31	24.5	46.4
% by weight of P <sub>2</sub> O <sub>5</sub>	21	25	29.5	28	16.5	0
% by weight of K <sub>2</sub> O	0	0	0	0	16.5	0
% by weight of H <sub>2</sub> O	0.2	0.12	0.11	0.2	0.16	0.15
breaking strength (kg/cm <sup>2</sup> )						
measured directly	45	40	40	40	55	20
measured after 12 weeks	45	45	40	55	55	20

As stated above during the experiments, the residence time measured from mixing to prilling was about 1.2 secs at a load of 120 kg/hr. In order to investigate what is the

maximum allowable residence time for a reasonably prillable melt still to be obtained, a series of buffer vessels of increasing capacity were placed under the mixer to provide

increasing residence time. It appeared that after 4 secs a readily prillable melt was obtained. After a contact time in excess of 4 secs, there was increasing froth formation but the mixture remained prillable. As a result of the froth formation however the capacity of the prilling bucket is rapidly reduced. The mixing time (including the prilling step) should therefore, preferably be less than 6 secs.

WHAT WE CLAIM IS:—

1. A process for prilling a urea melt containing monoammonium phosphate, wherein the urea melt is mixed with solid monoammonium phosphate less than 10 seconds

before effecting prilling of the melt.

2. A process according to Claim 1, wherein the said mixing time is less than 6 seconds.

3. A process according to Claim 1 or Claim 2, wherein at least a part of the monoammonium phosphate used consists of monoammonium phosphate obtained from phosphoric acid prepared by the wet method.

4. A process according to Claim 1, substantially as hereinbefore described with particular reference to the Examples.

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